[F1ANNEX II

GENERAL PROVISIONS ON METHODS OF ANALYSIS FOR FEED

Textual Amendments

F1 Substituted by Commission Regulation (EU) No 691/2013 of 19 July 2013 amending Regulation (EC) No 152/2009 as regards methods of sampling and analysis (Text with EEA relevance).

A.PREPARATION OF SAMPLES FOR ANALYSIS

1. Purpose

The procedures described below concern the preparation for analysis of samples, sent to the control laboratories after sampling in accordance with the provisions laid down in Annex I.

The laboratory samples must be prepared in such a way that the amounts weighed out, as provided for in the methods of analysis, are homogeneous and representative of the final samples.

2. Precautions to be taken

The sample preparation procedure to be followed is dependent on the methods of analysis to be used and the constituents or substances to be controlled. It is therefore of major importance that it is ensured that the followed sample preparation procedure is appropriate for the used method of analysis and for constituents or substances to be controlled.

All the necessary operations must be performed in such a way as to avoid as far as possible contamination of the sample and changes of its composition.

Grinding, mixing and sieving shall be carried out without delay with minimal exposure of the sample to the air and light. Mills and grinders likely to appreciably heat the sample shall not be used.

Manual grinding is recommended for feed which are particularly sensitive to heat. Care shall also be taken to ensure that the apparatus itself is not a source of contamination.

If the preparation cannot be carried out without significant changes in the moisture content of the sample, determine the moisture content before and after preparation according to the method laid down in Part A of Annex III.

3. Procedure

3.1. *General procedure*

The test aliquot is taken from the final sample. Coning and quartering is not recommended because this might provide test aliquots with high splitting error.

3.1.1. *Feed which can be ground as such*

— Mix the sieved final sample and collect it in a suitable clean, dry container fitted with an air-tight stopper. Mix again in order to ensure full homogenisation, immediately before weighing out the amount for analysis (test aliquot).

3.1.2. *Feed which can be ground after drying*

— Unless otherwise specified in the methods of analysis, dry the final sample to bring its moisture content down to a level of 8 to 12 %, according to the preliminary

drying procedure described under point 4.3 of the method of determination of moisture mentioned in Part A of Annex III). Then proceed as indicated in section 3.1.1.

3.1.3. *Liquid or semi-liquid feed*

— Collect the final sample in a suitable clean, dry container, fitted with an air-tight stopper. Mix thoroughly in order to ensure full homogenisation immediately before weighing out the amount for analysis (test aliquot).

3.1.4. *Other feed*

— Final samples which cannot be prepared according to one of the above procedures shall be treated by any other procedure which ensures that the amounts weighed out for the analysis (test aliquot) are homogeneous and representative of the final samples.

3.2. *Specific procedure in case of examination by visual inspection or by microscopy or in cases where the whole aggregate sample is homogenised*

— In case of an examination by visual inspection (without making use of microscope), the whole laboratory sample is used for examination.

— In case of a microscopic examination, the laboratory may reduce the aggregate sample, or further reduce the reduced sample. The final samples for defence and eventually reference purposes are taken following a procedure equivalent to the procedure followed for the final sample for enforcement.

— In case the whole aggregate sample is homogenized, the final samples are taken from the homogenized aggregate sample.

4. **Storage of samples**

Samples must be stored at a temperature that will not alter their composition. Samples intended for the analysis of vitamins or substances which are particularly sensitive to light shall be stored in such conditions that the sample is not adversely affected by light.

B. PROVISIONS RELATING TO REAGENTS AND APPARATUS USED IN METHODS OF ANALYSIS

1. Unless otherwise specified in the methods of analysis, all analytical reagents must be analytically pure (a.p.). When trace analysis is carried out, the purity of the reagents must be checked by a blank test. Depending upon the results obtained, further purification of the reagents may be required.

2. Any operation involving preparation of solutions, dilution, rinsing or washing, mentioned in the methods of analysis without indication as to the nature of the solvent or diluent employed, implies that water must be used. As a general rule, water shall be demineralised or distilled. In particular cases, which are indicated in the methods of analysis, it must be submitted to special procedures of purification.

3. In view of the equipment normally found in control laboratories, only those instruments and apparatus which are special or require specific usage are referred to in the methods of analysis. They must be clean, especially when very small amounts of substances have to be determined.

C. APPLICATION OF METHODS OF ANALYSIS AND EXPRESSION OF THE RESULTS

1. **Extraction procedure**

Several methods determine a specific extraction procedure. As a general rule, other extraction procedures than the procedure referred to in the method can be applied on the condition that the

used extraction procedure has been proven to have the equivalent extraction efficiency for the matrix analysed as the procedure mentioned in the method.

2. **Clean-up procedure**

Several methods determine a specific clean-up procedure. As a general rule, other clean-up procedures than the procedure referred to in the method can be applied on the condition that the used clean-up procedure has been proven to result in equivalent analytical results for the matrix analysed as the procedure mentioned in the method.

3. **Number of determinations**

In case of the analysis of undesirable substances, if the result of the first determination is significantly (> 50 %) lower than the specification to be controlled, no additional determinations are necessary, on the condition that the appropriate quality procedures are applied. In other cases a duplicate analysis (second determination) is necessary to exclude the possibility of internal cross-contamination or an accidental mix-up of samples. The mean of the two determinations, taking into account the measurement uncertainty is used for verification of compliance.

In case of the control of the declared content of a substance or ingredient, if the result of the first determination confirms the declared content, i.e. the analytical result falls within the acceptable range of variation of the declared content, no additional determinations are necessary, on the condition that the appropriate quality procedures are applied. In other cases a duplicate analysis (second determination) is necessary to exclude the possibility of internal cross-contamination or an accidental mix-up of samples. The mean of the two determinations, taking into account the measurement uncertainty is used for verification of compliance.

In some cases this acceptable range of variation is defined in legislation such as in Regulation (EC) No 767/2009 of the European Parliament and of the Council of 13 July 2009 on the placing on the market and use of feed, amending European Parliament and Council Regulation (EC) No 1831/2003 and repealing Council Directive 79/373/EEC, Commission Directive 80/511/EEC, Council Directives 82/471/EEC, 83/228/EEC, 93/74/EEC, 93/113/EC and 96/25/EC and Commission Decision 2004/217/EC[(1)].

4. **Reporting of the method of analysis used**

The analysis report shall mention the method of analysis used.

5. **Reporting of the analytical result**

The analytical result shall be expressed in the manner laid down in the method of analysis to an appropriate number of significant figures and shall be corrected, if necessary, to the moisture content of the final sample prior to preparation.

6. **Measurement uncertainty and recovery rate in case of analysis of undesirable substances**

As regards undesirable substances within the meaning of Directive 2002/32/EC, a product intended for animal feed shall be considered as non-compliant with the established maximum content, if the analytical result, relative to a feed with a moisture content of 12 %, is deemed to exceed the maximum content taking into account expanded measurement uncertainty and correction for recovery. In order to assess compliance, the analysed concentration is used after being corrected for recovery and after deduction of the expanded measurement uncertainty. This procedure is only applicable in cases where the method of analysis enables the estimation of measurement uncertainty and correction for recovery (e.g. not possible in case of microscopic analysis).

The analytical result shall be reported as follows (in so far the used method of analysis enables to estimate the measurement uncertainty and recovery rate):

(a) corrected for recovery, the level of recovery being indicated. The correction for recovery is not necessary in case the recovery rate is between 90-110 %.

(b) as 'x +/– U', whereby x is the analytical result and U is the expanded measurement uncertainty, using a coverage factor of 2 which gives a level of confidence of approximately 95 %.

However, if the result of the analysis is significantly (> 50 %) lower than the specification to be controlled, and on the condition that the appropriate quality procedures are applied and the analysis serves only the purpose of checking compliance with legal provisions, the analytical result might be reported without correction for recovery and the reporting of the recovery rate and measurement uncertainty might be omitted in these cases.**]**

(1) **[**[F1]OJ L 229, 1.9.2009, p. 1.**]**

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Changes to legislation:
There are currently no known outstanding effects for the Commission Regulation (EC) No 152/2009, ANNEX II.